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526 Rec'd PCT/PTO 19 SEP 2000

FORM PTO-1590 (Modified)  
(REV 11-98)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371

15268.1

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR

09/646767

INTERNATIONAL APPLICATION NO.

PCT/DE99/00782

INTERNATIONAL FILING DATE

March 17, 1999

PRIORITY DATE CLAIMED

March 20, 1998

TITLE OF INVENTION

PROPELLANTS FOR GAS GENERATOR

APPLICANT(S) FOR DO/EO/US

Eduard Gast, Bernhard Schmid and Peter Semmler



Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371 (c) (2))
  - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☒ has been transmitted by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ A copy of the International Search Report (PCT/ISA/210).
8. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))
  - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☐ have been transmitted by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☒ have not been made and will not be made.
9. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
10. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).
11. ☒ A copy of the International Preliminary Examination Report (PCT/IPEA/409).
12. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).

## Items 13 to 20 below concern document(s) or information included:

13. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
14. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
15. ☒ A **FIRST** preliminary amendment.
16. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
17. ☐ A substitute specification.
18. ☐ A change of power of attorney and/or address letter.
19. ☒ Certificate of Mailing by Express Mail
20. ☒ Other items or information:

## Postcard

Check No. 117609 in the amount of \$840.00

Copy of the cover page of PCT/DE99/00782 (WO 99/48843)

English translation of claims 1-16 submitted under Article 34 subsequent to the Written

Opinion and prior to the International Preliminary Examination.

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR

INTERNATIONAL APPLICATION NO.

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09/646767

PCT/DE99/00782

15268.1

21. The following fees are submitted:

BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)) :

- ☐ Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO ..... \$970.00
- ☒ International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO ..... \$840.00
- ☐ International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... \$690.00
- ☐ International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... \$670.00
- ☐ International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4) ..... \$96.00

ENTER APPROPRIATE BASIC FEE AMOUNT =

\$840.00

Surcharge of \$130.00 for furnishing the oath or declaration later than months from the earliest claimed priority date (37 CFR 1.492 (e)).

☐ 20 ☐ 30

\$0.00

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	
Total claims	18 - 20 =	0	x \$18.00	\$0.00
Independent claims	1 - 3 =	0	x \$78.00	\$0.00
Multiple Dependent Claims (check if applicable).			<input type="checkbox"/>	\$0.00

TOTAL OF ABOVE CALCULATIONS =

\$840.00

Reduction of 1/2 for filing by small entity, if applicable. Verified Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28) (check if applicable).

☐

\$0.00

SUBTOTAL =

\$840.00

Processing fee of \$130.00 for furnishing the English translation later than months from the earliest claimed priority date (37 CFR 1.492 (f)).

☐ 20 ☐ 30

+

\$0.00

TOTAL NATIONAL FEE =

\$840.00

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) (check if applicable).

☒

\$40.00

TOTAL FEES ENCLOSED =

\$880.00

Amount to be:  
refunded  
charged

\$

\$

- ☒ A check in the amount of \$840.00 to cover the above fees is enclosed.
- ☐ Please charge my Deposit Account No. in the amount of to cover the above fees.  
A duplicate copy of this sheet is enclosed.
- ☒ The Commissioner is hereby authorized to charge any fees which may be required, or credit any overpayment to Deposit Account No. 23-3178 A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

John M. Guynn  
Attorney for Applicants  
Registration No. 36,153



022913

PATENT TRADEMARK OFFICE

SIGNATURE

John M. Guynn

NAME

36,153

REGISTRATION NUMBER

September 19, 2000

DATE

09/646767

EXPRESS MAIL LABEL NO. EL571477070US

PATENT APPLICATION

Docket No.: 15268.1

422 Rec'd PCT/PTO 19 SEP 2000

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of

Gast et al.

Serial No.:

§ 371 PCT/DE99/00782

Filed:

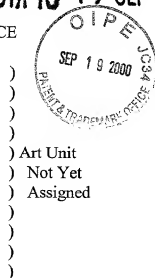
September 19 , 2000

For:

PROPELLANTS FOR GAS GENERATOR

Examiner:

Not Yet Assigned



PRELIMINARY AMENDMENT "A"

Assistant Commissioner for Patents  
Washington, D. C. 20231

Sir:

Preliminary to Examination of the above-identified application, please enter the following amendment:

IN THE SPECIFICATION:

At page 1, under the title "PROPELLANTS FOR GAS GENERATOR" please insert the following heading:

--BACKGROUND OF THE INVENTION--



At page 7, line 3, before the first full paragraph insert the following heading:

--SUMMARY OF THE INVENTION--

At page 7, line 21, between the paragraph ending in "generators." and the paragraph beginning with "According" please insert the following heading:

--DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS--

At page 9, line 21, after "Degussa AG." insert --The term "highly dispersed" is a literal translation of the German terms "hochdispers" which is a term of art that refers to very particular properties of the metal oxides referred to in the literature with respect to particle structure and particle size and wherein the "highly dispersed" metal oxides are prepared according to a specific process, *i.e.*, flame hydrolysis. Metal oxides prepared according to processes such as a wet process are not in "highly dispersed" form. The fact that the metal oxides prepared according to a wet process can optionally have a small particle size and can be present in finely divided form does not mean that these metal oxides are "highly dispersed" according to the present definition.--

IN THE CLAIMS:

Please amend the claims as follows:

1. (Amended) Propellant for gas generators, comprising

(a) at least one fuel selected from the group consisting of guanidine nitrate [(GUNI;  $\text{GuNO}_3$ )], dicyanamide, ammonium dicyanamide, sodium dicyanamide [(Na-DCA)], copper dicyanamide, tin dicyanamide, calcium dicyanamide [(Ca-DCA)], guanidine dicyanamide [(GDCA)], aminoguanidine bicarbonate [(AGB)], aminoguanidine nitrate [(AGN)], triaminoguanidine nitrate [(TAGN)], nitroguanidine [(NIGU)], dicyandiamide [(DCD)], azodicarbonamide, [(ADCA) as well as] tetrazole [(HTZ)], 5-aminotetrazole [(ATZ)], 5-nitro-1,2,4-triazole-3-on [(NTO)], salts and mixtures thereof[,];

(b) at least one of an alkali metal nitrate, [or] an alkaline earth metal nitrate, [or] ammonium nitrate, [-chlorate or -perchlorate,] an alkali metal chlorate, an alkaline earth metal chlorate, ammonium chlorate, an alkali metal perchlorate, an alkaline earth metal chlorate, or ammonium perchlorate, and

(c) at least one essentially chemically-inert slag trap with a high fusion point, [selected from the group comprising highly dispersed] said slag trap being at least one of  $\text{Al}_2\text{O}_3$  [having a specific surface of  $100 \pm 15 \text{ m}^2/\text{g}$ , highly dispersed]  $\text{TiO}_2$ , or [having a specific surface of  $50 \pm 15 \text{ m}^2/\text{g}$  and highly dispersed]  $\text{ZrO}_2$  [having a specific surface of  $40 \pm 10 \text{ m}^2/\text{g}$  and mixtures thereof] particles formed by a gas phase reaction so as to have a specific surface area of at least about  $40 \text{ m}^2/\text{g}$ .

2. (Amended) Propellant for gas generators according to claim 1, wherein component (a) is present in an amount of about 20 to 60 wt.-%, [preferably of about 28 to 52 wt.-% and in particular of about 45 to 51 wt.-%,] component (b) is present in an amount of about 38 to about 63 wt.-%, [preferably of about 38 to about 55 wt.-% and in particular of about 39 to 45 wt.-%,] and component (c) is present in an amount of about 5 to 22 wt.-%[, preferably of about 8 to 20 wt.-% and in particular of about 9 to 11 wt.-%].

3. (Amended) Propellant for gas generators according to claim 1 [or 2], wherein component (a) is selected from the group consisting of nitroguanidine, 5-aminotetrazole, dicyandiamide, dicyanamide, sodium- and calcium dicyanamide, [and] guanidine nitrate, and mixtures thereof.

4. (Amended) Propellant for gas generators according to [any one of] claim[s] 1 [to 3], wherein component (b) is selected from the group consisting of sodium-, potassium- and strontium nitrate.

5. (Amended) Propellant for gas generators according to [any one of] claim[s] 1 [to 4], wherein a [part] portion of the component (c) is a carrier for a platinum metal or a metal alloy of platinum metals or copper in a catalytic effective layer thickness.

6. (Amended) Propellant for gas generators according to claim 5, wherein the platinum metal is selected from ruthenium [(Ru)], [O]osmium [(Os)], rhodium [(Rh)], iridium [(Ir)], palladium [(Pd)] and platinum [(Pt)].

7. (Amended) Propellant for gas generators according to claim 5, wherein the metal alloy of platinum metals is [selected from] at least one of a Pt/Pd alloy [and] or a Pt/Rh alloy[s].

8. (Amended) Propellant for gas generators according to [any one of] claim[s] 5 [to 7], wherein the weight portion of the catalyst with respect to component (c) is 0.1 to 5 wt.-%, preferably 0.2 to 1.2 wt.-%].

9. (Amended) Propellant for gas generators according to [any one of] claim[s] 1 [to 8], wherein component (a) is nitroguanidine, component (b) is strontium nitrate and component (c) is highly dispersed  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$  or  $\text{ZrO}_2$ .

11. (Amended) Propellant for gas generators according to [any one of] claim[s] 1 [to 9], [containing in addition] further including a component (d) that is at least one slag former[,] selected from the group consisting of alkali metal [and] carbonates, alkaline earth metal carbonates, alkali metal oxides, [and] alkaline earth metal oxides, silicates, aluminates, alumin[i]um silicates, silicon nitride [ $(\text{Si}_3\text{N}_4)$ ] and iron(III)oxide.

13. (Amended) Propellant for gas generators according to [any one of] claim[s] 1 [to 12], further [containing] including a component (e) that is at least one binder being soluble in water at room temperature.

14. (Amended) Propellant for gas generators according to claim [13] 1, [wherein the] further including a component (e) that is at least one binder [is] selected from the group consisting of cellulose compounds, polymers of one or more polymeri[s]zable olefinic unsaturated monomers, a metal salt of stearic acid being insoluble in water at room temperature and graphite.

15. (Amended) Propellant for gas generators according to claim [13 or] 14, wherein the binder is present in an amount of 0 to 2 wt.-%[, preferably of 0.3 to 0.8 wt.-%].

16. (Amended) [Use of the p] Propellant for gas generators according to [any one of] claim[s] 1, [to 15] wherein the propellant is suitable for use as at least one of a gas-generating agent in airbags, [as] an extinguishing agent or [as] a propellant.



IN THE ABSTRACT:

Please cancel the existing abstract and replace it with the following abstract:

--Solid propellants for gas generators, wherein said propellants are mainly intended for use in propelling charges for gas generators used in airbags or seat-belt pre-tensioning devices on the basis of nitrogen-rich fuels having a low carbon content. The solid propellants also include an oxidizing agent as well as an essentially chemically-inert slag trap used to trap airborne particulates and molten slags generated during ignition of the solid propellants. The slag traps are characterized as having a high fusion point and a high specific surface area such that they are well suited for trapping molten slag and other particulates during ignition of the solid propellants. The slag traps typically comprise aluminum oxide, titanium oxide or zirconium oxide and are manufactured by a gas phase reaction (*e.g.*, flame hydrolysis) so as to be "highly dispersed" but with no pores and with no formation of defined agglomerates as is usually the case in the preparation of aluminum oxide, titanium oxide or zirconium oxide by a wet process.

REMARKS



The foregoing amendments were made to better conform the specification and claims to a style that is more acceptable for filing in the United States. The term "highly dispersed", which is a literal translation of the German term "hochdispers", was more particularly explained so as to reveal this term's true meaning as intended when the original parent German application and corresponding PCT application were drafted. No new matter was added since the definition of the term "hochdispers" is inherent subject matter. In addition, the claims were amended to more particularly claim what Applicants regard as their invention. Finally, Applicants have prepared a new abstract that is more description of the invention compared to the abstract set forth in the PCT application.

Dated this 19<sup>th</sup> day of September 2000.

Respectfully submitted,

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PROPELLANTS FOR GAS GENERATORS

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The present invention relates to solid propellants for gas generators (gas-generating mixtures), wherein said propellants are mainly intended for use in propelling charges for gas generators used in airbags or seat-belt pre-tensioning devices on the basis of nitrogen-rich fuels having a carbon content as low as possible. The solid propellants for gas generators further include an essentially chemically-inert slag trap in highly dispersed form which has a high fusion point, wherein said slag trap acts as an inner filter and substantially prevents the formation of powder (dust-type) particles as well as their exit from the housing of the gas generator.

Thus, the present invention relates to a process for trapping the liquid and solid burn-up products and dust-type slag particles, respectively, within the propelling charge for gas generators directly during the formation. It is thus possible to use a simple structured filter package within the housing of the gas generator.

The present invention further relates to the use of catalysts on the basis of platinum metals (Ru, Os, Rh, Ir, Pd, Pt) or metal alloys of platinum metals or copper on the slag traps as carriers in solid propellants for gas generators. In particular, it relates to the use in solid propelling charges for gas generators used in airbags.

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An airbag essentially comprises a housing of the gas generator filled with the propelling charges for gas generators, generally in tablet form, and an initial detonator (squib) for detonating the propelling charge for gas generators, and also a gas bag. Suitable detonators are disclosed, for example, in US 4,931,111. The gas bag, which is initially folded into a small bag, is filled, after the initial detonation, with the gases produced in the burn-up of the propelling charge for gas generators and reaches its full volume in a time period of about 10-50 ms. The exit of hot sparks, molten material and solids from the gas generator into the gas bag has to be largely prevented, since it could result in the destruction of the gas bag or in injury to the vehicle passengers. This is achieved by binding and filtering of the slag formed by burning up the propelling charge for gas generators.

Propelling charges for gas generators to be used in airbags on the basis of sodium azide are well known. However, the use of the highly toxic sodium azide requires a work intensive and costly process for the preparation of the propelling charges for gas generators. Furthermore, the worldwide ever increasing number of non-burned-up propelling charges for gas generators in used vehicles leads to a disposal and security problem.

Therefore, in recent years there have been efforts to find suitable substitutes for sodium azide.

DE-A-44 35 790 discloses propellants for gas generators on the basis of guanidine compounds on suitable carriers, which essentially exhibit an improved burn-up behaviour and an improved building of the slag. DE-A-44 35 790 does not give any hints as to the use of essentially chemically-inert slag traps in highly dispersed form which have a high fusion point

From EP-B-0 482 852 and the prior art referred to in this document azide-free propelling charges for gas generators, in particular for use in airbags, are known. The gas-generating mixture disclosed in EP-B-0 482 852 contains a) a fuel selected from aminotetrazole, tetrazole, bitetrazole and metal salts thereof and triazole compounds and metal salts thereof; b) an oxygen containing oxidizing agent selected from nitrates and perchlorates of alkaline metals, alkaline earth metals, lanthanoides and ammonium, and chlorates and peroxides of alkaline metals and alkaline earth metals; and either c) a high temperature slag forming material selected from alkaline earth metal oxides, -hydroxides, -carbonates, -oxalates, -peroxides, -nitrates, -chlorates and -perchlorates and alkaline earth metal salts of tetrazoles, bitetrazoles and triazoles, and d) a low temperature slag forming material selected from silicon dioxide, boron oxide, vanadium pentoxide, naturally occurring clays and talcum, alkalimetal silicates, -borates, -carbonates, -nitrates, -perchlorates and -chlorates and alkalimetal salts of tetrazoles, bitetrazoles and triazoles; or e) a high temperature slag forming material selected from transition metal oxides, -hydroxides, -carbonates, -oxalates, -peroxides, -nitrates, -chlorates and -perchlorates; and f) a low temperature slag forming material which is silicon dioxide, wherein the amount of d) or f) is sufficient to achieve the formation of a coherent mass or slag, however, is not large enough to build a liquid with low viscosity. It is evident that a single material can be used in more than one category.

The essential advantage of a propelling charge for gas generators of this kind is the advantageous formation of a slag

However, disadvantages of this kind of propelling charges for gas generators are as follows:

EP-B-0 482 852 does not give any hints as to how these problems can be solved by modifying the composition of the propelling charge for gas generators.

However, the teaching of US 4,948,439 does not provide any suggestion as to how the portion of toxic gaseous burn-up products developed during the burning up of propelling charges for gas generators which contain as fuel tetrazole or triazole compounds, metal salts or mixtures thereof, can be reduced. On the contrary, a process for inflating an airbag is described according to which a primary gas mixture is initially obtained by ignition of a propelling charge for gas generators which contains as a fuel at least one tetrazole or

However, the mixing with the surrounding atmosphere leads to a more complex total airbag system (size, structure, etc.). A problem is the rate with which the airbag has to be inflated (10-50 ms) in case that in addition surrounding atmosphere has to be drawn in.

DE-C-44 01 213 discloses gas-generating mixtures comprising a fuel, an oxidizing agent, a "catalyst" and a cooling agent, characterized in that the oxidizing agent is  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{Cu}(\text{OH})_2$  and the catalyst is a metal oxide or a mixture of metal oxides or a metal mixed oxide.

In addition, DE-C-44 01 214 discloses gas-generating mixtures having a similar composition, in which the catalyst is a metal or metal alloy, preferably a pyrophoric metal or a pyrophoric metal alloy on a carrier. The carrier is a silicate, preferably a layer silicate or framework silicate. As a metal Ag is in particular useful. Among the known fuels used are triaminoguanidine nitrate (TAGN), nitroguanidine (NIGU, NQ), 3-nitro-1,2,3-triazole-5-on and, in particular, diguanidine-5,5'-azotetrazolate (GZT).

The most important advantage of the gas-generating mixtures described in the two German patents mentioned above is according to the teaching in these patents the decrease in the burn-up temperature and the increase in the burn-up rate.

The gas-generating mixtures described in DE-C-44 01 213 and DE-C-44 01 214 do not contain low temperature melting and

high temperature melting slag formers and no slag traps according to the present invention. On the contrary, it is said that slag formers are not necessary.

Contrary to this allegation, the present inventors found that the use of low temperature melting and high temperature melting slag formers, in particular the use of the slag traps according to the present invention results in a significant reduction of toxic gaseous burn-up products. A part of the high temperature melting slag trap can be used as a carrier for a platinum metal and for a metal alloy consisting of platinum metals and, therefore, can function as a part of the catalyst.

In both of the above mentioned German patents the term "catalyst" is used in an extended sense and is an active part of the reaction, which "catalyst" can be reacted and acts to direct the reaction and/or to accelerate the reaction.

Therefore, it is not a catalyst in the strict sense of the word, since a catalyst is not a component to be reacted in a reaction. A catalyst in the strict sense of the word is not consumed during a reaction, i.e. is not reacted.

The definition of a catalyst further comprises that the catalyst is added to the reaction mixture only in a very low concentration. However, in both German patents the portion of the "catalyst" in that gas-generating mixture is up to 30% by weight and is therefore an essential part of the gas-generating mixture, also as regards its proportion in the mixture.

Thus, in summary, it is true that in DE-C-44 01 213 and DE-C-44 01 214 the term "catalyst" is used but the meaning of this



term does not correspond to the common definition of a catalyst, as is also indicated in the two patents themselves.

The technical problem underlying the present invention vis-à-vis the prior art is to provide improved propellants for gas generators, in particular for airbags, the burn-up behaviour of which can be adjusted as desired and which minimize in particular the formation of toxic gases and of powder (dust-type) components which could exit from the housing of the gas generator and enter the lungs.

The propelling charges for gas generators prepared from the propellants for gas generators are intended to be thermally stable, readily ignitable, fast-burning - even at low temperature, and well storable and ensure a high gas yield. In addition, said propellants for gas generators are intended to make it possible to reduce the size of the housing of the gas generator and the number of components and simplify the housing of the gas generator and consequently reduce its weight compared with known generators.

According to the present invention, these objects are achieved by a propellant for gas generators, comprising

(A) at least one fuel selected from the group comprising guanidine nitrate (GUNI;  $\text{GuNO}_3$ ), dicyanamide, ammonium dicyanamide, sodium dicyanamide (Na-DCA), copper dicyanamide, tin dicyanamide, calcium dicyanamide (Ca-DCA), guanidine dicyanamide (GDCA), aminoguanidine bicarbonate (AGB), aminoguanidine nitrate (AGN), triaminoguanidine nitrate (TAGN), nitroguanidine (NIGU), dicyandiamide (DCD), azodicarbonamide (ADCA) as well as tetrazole (HTZ), 5-aminotetrazole (ATZ), 5-nitro-1,2,4-triazole-3-on (NTO), salts and mixtures thereof,

(B) at least one alkali metal nitrate or alkaline earth metal nitrate or ammonium nitrate, -chlorate or -perchlorate,

(C) at least one essentially chemically-inert slag trap which has a high fusion point selected from the group comprising  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$  and  $\text{ZrO}_2$  in highly dispersed form and mixtures thereof, and

optionally (D) at least one slag former selected from alkali metal and alkaline earth metal carbonates and -oxides, silicates, aluminates and aluminumsilicates, iron(III)oxide as well as silicon nitride ( $\text{Si}_3\text{N}_4$ ), which results during burn-up in the formation of nitrogen ( $\text{N}_2$ ) and silicon dioxide ( $\text{SiO}_2$ ) for the further reaction and

optionally (E) at least one binder being soluble in water at room temperature.

Preferred fuels, component (A), are nitroguanidine (NIGU), 5-aminotetrazole (ATZ), dicyandiamide (DCD), dicyanamide, the salts thereof, in particular sodium- and calcium dicyanamide and guanidine nitrate, and mixtures thereof. These are virtually non-toxic, non-hygroscopic, sparsely soluble in water, thermally stable, burning even at low temperature and have a low impact and friction sensitivity. The gas yield during burn-up is high, wherein a large portion of nitrogen gas is developed.

Alkali metal salts (Li, Na, K) and alkaline earth metal salts (Mg, Ca, Sr, Ba) are examples for suitable salts of 5-aminotetrazole.

As oxidizing agents, component (B), alkali metal- or alkaline earth metal nitrates (such as lithium nitrate, sodium nitrate, potassium nitrate, magnesium nitrate, calcium nitrate,

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strontium nitrate or barium nitrate), ammonium nitrate, alkali metal- or alkaline earth metal chlorates or perchlorates (such as lithium-, sodium-, potassium-, magnesium-, calcium-, strontium- or barium chlorate and lithium-, sodium-, potassium-, magnesium-, calcium-, strontium- or barium perchlorate) as well as ammonium perchlorate and mixtures thereof can be used. Potassium nitrate and strontium nitrate are preferred. Strontium nitrate is non-hygroscopic, non-toxic and allows a high gas yield during burn-up. Potassium nitrate, in addition, has a low burn-up temperature.

Examples for essentially chemically-inert slag traps which have a high fusion point, component (C), are  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$  and  $\text{ZrO}_2$  in highly dispersed form or mixtures thereof.  $\text{Al}_2\text{O}_3$  having a BET-surface (according to DIN 66131) of  $100 \pm 15 \text{ m}^2/\text{g}$  (fusion point about  $2,050^\circ\text{C}$ ),  $\text{TiO}_2$  having a BET-surface of  $50 \pm 15 \text{ m}^2/\text{g}$  (fusion point about  $1,850^\circ\text{C}$ ) and  $\text{ZrO}_2$  having a BET-surface of  $40 \pm 10 \text{ m}^2/\text{g}$  (fusion point about  $2,700^\circ\text{C}$ ) are particularly preferred. These highly dispersed oxides are commercially available for example under the trade names Aluminiumoxid C, Titanoxid P25 and VP Zirkonoxid of Degussa AG.

These pyrogenic oxides are obtained by the reaction of the respective metal chloride with  $H_2$  and  $O_2$  in the corresponding molar ratio by gas phase reaction (flame hydrolysis). These oxides have no pores and no defined agglomerates as is usually the case in the preparation by a wet process.

The term "slag trap", component (C), according to the present invention refers to an essentially chemically-inert metal oxide which has a high fusion point and is in highly dispersed form, i.e. these oxides have in comparison to oxides in conventional form a much higher surface.

For example, conventional  $\text{Al}_2\text{O}_3$  as  $\alpha$ -oxide has a BET-surface of only 5-10  $\text{m}^2/\text{g}$ , conventional pigment- $\text{TiO}_2$  has a BET-surface of only 5-10  $\text{m}^2/\text{g}$  and conventional  $\text{ZrO}_2$  has a BET-surface of only 3-8  $\text{m}^2/\text{g}$  (for fire-resistant products), whereas the metal oxides used in the propelling charges for gas generators according to the present invention have a BET-surface of about 40 up to about 100  $\text{m}^2/\text{g}$ , particularly preferred of about 50 to about 100  $\text{m}^2/\text{g}$  and, in particular, of about 100  $\text{m}^2/\text{g}$ .

Furthermore, the slag traps according to the present invention are characterized by their high fusion point of about 1,850 to about 2,700°C. As a consequence of these high fusion points, the slag traps do not melt during the reaction and, thus, function as solids.

Furthermore, the slag traps according to the present invention are essentially chemically-inert compounds, i.e. the slag traps according to the present invention do not take part in chemical reactions during the burn-up reaction of the propelling charges for gas generators or do take part only to a small degree on the surface of the metal oxides used as a slag trap. The highly resolved lattices, i.e. the large inner surface of for example  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$  or  $\text{ZrO}_2$  (in highly dispersed form) cause on the one hand cooling of the burn-up products due to their inactivity and causes on the other hand take-up of, in particular, liquid and/or solid slag portions and particles, respectively, which are developed during burn-up. Thus, the tablet form of the propelling charges for gas generators remains during and after burn-up and possibly formed fragments and pieces can be easily filtered. That means, almost no dust is formed which could exit from the propelling charges for gas generators and consequently from the housing of the gas generators during burn-up. Thus, the

slag traps function as an internal filter in the propelling charges for gas generators themselves and, thus, substantially prevent the formation and exit of dust-type slag portions from the housing of the gas generator. Accordingly, an essential simplification of the filter of the housing of the gas generator is obtained, in that additional (mechanical) fine filters in the housing of the gas generator are in part not necessary. This also leads to an advantageous saving of weight of the airbag gas generator.

Simultaneously, the formation of dust-type particles which could exit the gas generator of an airbag and could enter the lungs is minimized by the formation of slags. Dust-type particles which can enter the lungs have a diameter of about 6  $\mu\text{m}$  or less.

Optionally, slag formers, component (D), such as alkali metal and alkaline earth metal carbonates (e.g. sodium carbonate, potassium carbonate, magnesium carbonate, calcium carbonate, strontium carbonate and barium carbonate), alkali metal or alkaline earth metal oxides (e.g. sodium-, potassium-, magnesium-, calcium-, strontium- and barium oxide), silicates (e.g. hectorite), aluminates (e.g. sodium-beta-aluminate ( $\text{Na}_2\text{O}_{11}\text{Al}_2\text{O}_3$ ) or tricalcium aluminate ( $\text{Ca}_3\text{Al}_2\text{O}_6$ )) or aluminum silicates (e.g. bentonites or zeolithes) or iron(III)oxide or mixtures thereof can be used.

The function of component (D) is to form an easily filterable slag during the burn-up of the propellant for gas generators.

In addition, the slag formers, component (D), can serve as a coolant. The silicates, aluminates and aluminum silicates react with the alkali metal and alkaline earth metal oxides which are formed during burn-up.

The present invention further relates to the use of catalysts on the basis of platinum metals (Ru, Os, Rh, Ir, Pd, Pt) or metal alloys of platinum metals or copper on highly dispersed slag traps as carriers in the solid propellants for gas generators of the present invention, in particular for use in solid propelling charges for gas generators for airbags.

A part of the slag trap, component (C), can serve as a carrier for a platinum metal or a metal alloy of platinum metals or copper in a catalytic effective layer thickness.

Platinum metals are ruthenium (Ru), osmium (Os), rhodium (Rh), iridium (Ir), palladium (Pd) and platinum (Pt). The catalysts used in the present invention are preferably based on Rh, Pd or Pt and, in particular, on Pt.

Examples for metal alloys of platinum metals are all catalytically effective metal alloys of the platinum metals mentioned above, preferably Pt/Pd and Pt/Rh alloys.

The metals or metal alloys of platinum metals are on the carrier in a catalytically effective layer thickness, preferably in a monolayer.

The catalysts are contained in the propelling charges for gas generators only in catalytic amounts. The weight portion with respect to component (C) is 0.1-5 % by weight, preferably 0.2-1.2 % by weight of component (C).

Preferred catalysts are those having  $\text{Al}_2\text{O}_3$  as the highly dispersed carrier and Pt, Pd or Cu, in particular Pt, as metal.

Suitable catalysts are obtainable from Degussa AG, e.g. 1% Pt on  $\gamma\text{-Al}_2\text{O}_3$  or 1% Pd+Pt on  $\gamma\text{-Al}_2\text{O}_3$ .

The catalysts are useful to direct the reaction in that almost no toxic gaseous burn-up products such as carbon monoxide (CO), nitrogen oxides (NO<sub>x</sub>) and ammonia (NH<sub>3</sub>) are formed.

The above mentioned catalysts are in particular useful for the use in propelling charges for gas generators in airbags.

In addition to the advantages which result from the use of highly dispersed metal oxides (i.e. decrease of solid dust-type particles, i.e. of coarse and fine dust) the anyhow low portion of toxic gases is even more reduced.

The catalysts can be recycled by known processes from released, i.e. used airbags, as well as from non-released, i.e. airbags from used vehicles. This results in the reduction of waste which could pollute the environment and allows the re-use of the catalyst metals. The catalyst metal and metal alloy, respectively, is not oxidized during burn-up.

The catalyst must not be added as an additional part of the propelling charge for gas generators but the catalyst is part of a component (component (C)) being present anyhow in the propelling charge for gas generators.

Component (A) is present in an amount of about 20 to 60 wt.-%, preferably from about 28 to 52 wt.-%, and in particular from about 45 to 51 wt.-%; component (B) is present in an amount of about 38 to about 63 wt.-%, preferably from about 38 to about 55 wt.-%, and in particular from about 39 to 45 wt.-%; component (C) is present in an amount from about 5 to 22 wt.-%, preferably from about 8 to 20 wt.-%, and in particular from about 9 to 11 wt.-%; finally, component (D), in case it is present at all, is present in an amount of about 2 to 12 wt.-%, preferably from about 4 to 10 wt.-%. All the wt.-%

given refer to the total composition of the propelling charge for gas generators.

Optionally, the propellant for gas generators may further contain a binder being soluble in water at room temperature as component (E). Preferred binders are cellulose compounds or polymers of one or more polymerizable olefinic unsaturated monomers. Examples of cellulose compounds are cellulose ethers, such as carboxymethylcellulose, methylcelluloseether, in particular methylhydroxyethylcellulose. A methylhydroxyethylcellulose which can be used satisfactorily is CULMINAL® MHEC 30000 PR supplied by the company Aqualon. Suitable polymers having binding action are polyvinylpyrrolidone, polyvinylacetate, polyvinylalcohol and polyvinylbutyral, for example Pioloform® B supplied by the company Wacker Chemie, Burghausen, Germany.

Further, metal salts of stearic acid being insoluble in water at room temperature, such as aluminum stearate, magnesium stearate, calcium stearate or zinc stearate, can be used as a binder (component (E)).

Furthermore, graphite is suitable as a binder.

Component (E) is present in an amount of 0 to 2 wt.-% and preferably from 0.3 to 0.8 wt.-%.

The binder, component (E), serves as desensitizing agent and as processing aid in the production of granular material or tablets (pellets) from the propellant for gas generators. It furthermore serves to reduce the hydrophilic nature of the propelling charges for gas generators and to stabilize them.



Working Example:

The preparation of the propellants for gas generators (Examples 1 to 57 of Table I) and of the propelling charges for gas generators was carried out as described in the following:

The roughly premixed raw materials (components (A), (B), (C) and optionally (D) and (E)) have been milled and predensified, respectively, by means of a ball mill.

The granulation of the propellant mixtures for gas generators has been carried out in a vertical mixer in that about 20 % of water has been added during steering and at an elevated temperature of about 40°C. After a short venting the obtained mixed mass has been grinded at room temperature through a grinding machine having a 1 mm-sieve. The thus obtained granulate has been dried for about two hours at 80°C in a drying oven. The ready-to-use granulate of the propellant for gas generators (grain-(size)distribution 0-1 mm) has been compressed into tablets (pellets) using a rotary pelleting machine. These pellets for the propelling charge for gas generators have been dried again at 80°C in a drying oven.

The tablets and pellets, respectively, used in the gas generators and prepared from the propellants for gas generators can be prepared according to processes known in the art, for example by hot press working, extruding, in rotary-pelleting (compression) machines or tableting machines. The size of the pellets and tablets, respectively, depends on the desired burning time in each individual case.

The propellant for gas generators according to the present invention consists of non-toxic, inexpensive components which can be easily prepared and the processing of which is not problematic. The components which are more expensive, i.e.

the catalyst metal, can be recycled according to known procedures. Due to the thermal stability of the components a good storability is obtained. The mixtures are readily ignitable. They are fast-burning and ensure high gas yields with a very low proportion of CO, NO<sub>x</sub> and NH<sub>3</sub> which proportion is below the admissible upper limit. Therefore, the mixtures according to the present invention are in particular suitable as gas-generating agents in the various airbag systems, as extinguishing agents or propellants.

The following examples 1 to 57 illustrate but do not limit the present invention. Examples 15, 18 and 21 are comparative examples in which conventional ZrO<sub>2</sub>, TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> has been used.

Table I:

The superscripts of the table have the following meaning:

- 1     Titandioxid P25, supplied by Degussa AG
- 2     Zirkonoxid VP, supplied by Degussa AG
- 3     Aluminiumoxid C, supplied by Degussa AG
- 4     Titandioxid Kronos 3025, supplied by Kronos Titan-GmbH
- 5     Zirkonoxid, supplied by Merck
- 6     Aluminiumoxid NO 615-30 II 24, supplied by Nabaltec
- 7     Oxidation catalyst 1% Pt on Gamma-Aluminiumoxid, supplied by Degussa AG
- 8     Oxidation catalyst 1% Pd+Pt on Gamma-Aluminiumoxid, supplied by Degussa AG
- 9     Ironoxide, Bayoxide E8710, supplied by Bayer AG
- 10    Bentone EW, supplied by Rheox, Inc.
- 11    CULMINAL MHEC 30000 PR, supplied by Aqualon

### Table I

Example No.		1	2	3	4	5	6
A = ATZ	[%]	30,2	32,8	29,75	29,7	29,75	29,7
NIGU	[%]	-	-	-	-	-	-
Ca-DCA	[%]	-	-	-	-	-	-
Na-DCA	[%]	-	-	-	-	-	-
TAGN	[%]	-	-	-	-	-	-
GuNO <sub>3</sub>	[%]	-	-	-	-	-	-
B = KNO <sub>3</sub>	[%]	49,8	-	50,25	-	50,25	-
Sr(NO <sub>3</sub> ) <sub>2</sub>	[%]	-	57,2	-	54,8	-	54,8
NaNO <sub>3</sub>	[%]	-	-	-	-	-	-
C = TiO <sub>2</sub> <sup>1</sup>	[%]	-	-	20,0	15,0	-	-
ZrO <sub>2</sub> <sup>2</sup>	[%]	-	-	-	-	20,0	15,0
Al <sub>2</sub> O <sub>3</sub> <sup>3</sup>	[%]	10,0	10,0	-	-	-	-
Al <sub>2</sub> O <sub>3</sub> + 1%Pt <sup>7</sup>	[%]	-	-	-	-	-	-
Al <sub>2</sub> O <sub>3</sub> + 1%(Pd+Pt) <sup>8</sup>	[%]	-	-	-	-	-	-
D = Iron(III)oxide <sup>9</sup>	[%]	10,0	-	-	-	-	-
Aluminiumsilicate <sup>10</sup>	[%]	-	-	-	-	-	-
Siliconnitride Si <sub>3</sub> N <sub>4</sub>	[%]	-	-	-	-	-	-
E = Graphite	[%]	-	-	-	-	-	-
Methylhydroxyethylcellulose <sup>11</sup>	[%]	-	-	-	-	-	-
Polvinylbutrale	[%]	-	-	-	0,5	-	0,5
Calculated Values:							
Gas yield (V=constant)	[mol/kg]	17,8	19,3	17,6	21,7	17,6	18,0
Temperature (p=135*10 <sup>5</sup> Pa)	[K]	1780	2420	1780	2370	1780	2520
Measured Values (in 60 dm <sup>3</sup> can):							
Carbonmonoxide	[ppm]	4000	2800	3000	3300	3000	3300
Nitrogenoxide	[ppm]	150	300	200	350	200	250
Ammonia	[ppm]	150	0	0	0	100	100
Coarse dust in the can	[g]	1,2	0,6	1,2	1,0	1,1	1,2
Fine dust in the can	[g]	0,2	0,1	0,3	0,3	0,3	0,3

Example No.		7	8	9	10	11	12
A = ATZ	[%]	29,75	32,8	29,75	32,8	21,5	25,6
NIGU	[%]	-	-	-	-	-	-
Ca-DCA	[%]	-	-	-	-	-	-
Na-DCA	[%]	-	-	-	-	-	-
TAGN	[%]	-	-	-	-	-	-
GuNO <sub>3</sub>	[%]	-	-	-	-	-	-
B = KNO <sub>3</sub>	[%]	50,25	-	50,25	-	58,0	-
Sr(NO <sub>3</sub> ) <sub>2</sub>	[%]	-	57,2	-	57,2	-	54,1
NaNO <sub>3</sub>	[%]	-	-	-	-	-	-
C = TiO <sub>2</sub> <sup>1</sup>	[%]	-	-	-	-	-	-
ZrO <sub>2</sub> <sup>2</sup>	[%]	-	-	-	-	-	-
Al <sub>2</sub> O <sub>3</sub> <sup>3</sup>	[%]	10,0	-	10,0	-	10,0	10,0
Al <sub>2</sub> O <sub>3</sub> + 1%Pt <sup>7</sup>	[%]	10,0	10,0	-	-	-	-
Al <sub>2</sub> O <sub>3</sub> + 1%(Pd+Pt) <sup>8</sup>	[%]	-	-	10,0	10,0	-	-
D = Iron(III)oxide <sup>9</sup>	[%]	-	-	-	-	-	5,0
Aluminiumsilicate <sup>10</sup>	[%]	-	-	-	-	-	-
Siliconnitride Si <sub>3</sub> N <sub>4</sub>	[%]	-	-	-	-	10,0	5,0
E = Graphite	[%]	-	-	-	-	0,5	-
Methylhydroxyethylcellulose <sup>11</sup>	[%]	-	-	-	-	-	-
Polyvinylbutyrale	[%]	-	-	-	-	-	0,3
Calculated Values:							
Gas yield (V=constant)	[mol/kg]	17,6	19,3	17,6	19,3	16,8	16,8
Temperature (p=135*10 <sup>5</sup> Pa)	[K]	1780	2420	1780	2420	2120	2420
Measured Values (in 60 dm <sup>3</sup> can):							
Carbonmonoxide	[ppm]	2500	2300	2300	2100	4500	4000
Nitrogenoxide	[ppm]	200	250	200	250	400	250
Ammonia	[ppm]	0	0	0	0	200	150
Coarse dust in the can	[g]	0,7	0,6	0,7	0,7	0,9	1,3
Fine dust in the can	[g]	0,2	0,2	0,2	0,1	0,3	0,5

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Example No.		13	14	15	16	17	18
A = ATZ	[%]	-	-	-	-	-	-
NIGU	[%]	48,2	47,0	47,0	48,5	47,0	47,0
Ca-DCA	[%]	-	-	-	-	-	-
Na-DCA	[%]	-	-	-	-	-	-
TAGN	[%]	-	-	-	-	-	-
GuNO <sub>3</sub>	[%]	-	-	-	-	-	-
B = KNO <sub>3</sub>	[%]	41,3	-	-	41,0	-	-
Sr(NO <sub>3</sub> ) <sub>2</sub>	[%]	-	42,5	42,5	-	42,5	42,5
NaNO <sub>3</sub>	[%]	-	-	-	-	-	-
C = TiO <sub>2</sub> <sup>1 or 4</sup>	[%]	10,0 <sup>1</sup>	10,0 <sup>1</sup>	10,0 <sup>4</sup>	-	-	-
ZrO <sub>2</sub> <sup>2 or 5</sup>	[%]	-	-	-	10,0 <sup>2</sup>	10,0 <sup>2</sup>	10,0 <sup>5</sup>
Al <sub>2</sub> O <sub>3</sub> <sup>3</sup>	[%]	-	-	-	-	-	-
Al <sub>2</sub> O <sub>3</sub> + 1%Pt <sup>7</sup>	[%]	-	-	-	-	-	-
Al <sub>2</sub> O <sub>3</sub> + 1%(Pd+Pt) <sup>8</sup>	[%]	-	-	-	-	-	-
D = Iron(III)oxide <sup>9</sup>	[%]	-	-	-	-	-	-
Aluminiumsilicate <sup>10</sup>	[%]	-	-	-	-	-	-
Siliconnitride Si <sub>3</sub> N <sub>4</sub>	[%]	-	-	-	-	-	-
E = Graphite	[%]	-	0,5	0,5	-	0,5	0,5
Methylhydroxyethylcellulose <sup>11</sup>	[%]	-	-	-	-	-	-
Polyvinylbutyral	[%]	0,5	-	-	0,5	-	-
Calculated Values:							
Gas yield (V=constant)	[mol/kg]	23,8	23,1	23,1	23,9	23,1	23,1
Temperature (p=135*10 <sup>5</sup> Pa)	[K]	2030	2490	2490	2080	2550	2550
Measured Values (in 60 dm <sup>3</sup> can):							
Carbonmonoxide	[ppm]	8000	6500	8000	6500	6500	8000
Nitrogenoxide	[ppm]	600	450	450	800	700	800
Ammonia	[ppm]	100	0	0	150	0	0
Coarse dust in the can	[g]	1,4	0,3	0,7	1,0	0,1	0,3
Fine dust in the can	[g]	0,6	0,4	0,3	0,3	0,3	0,3

Example No.		19	20	21	22	23	24
A = ATZ	[%]	-	-	-	-	-	-
NIGU	[%]	50,6	46,0	46,0	46,5	50,6	46,5
Ca-DCA	[%]	-	-	-	-	-	-
Na-DCA	[%]	-	-	-	-	-	-
TAGN	[%]	-	-	-	-	-	-
GuNO <sub>3</sub>	[%]	-	-	-	-	-	-
B = KNO <sub>3</sub>	[%]	39,4	-	-	-	39,4	-
Sr(NO <sub>3</sub> ) <sub>2</sub>	[%]	-	43,5	43,5	38,5	-	38,5
NaNO <sub>3</sub>	[%]	-	-	-	-	-	-
C = TiO <sub>2</sub> <sup>1</sup>	[%]	-	-	-	-	-	-
ZrO <sub>2</sub> <sup>2</sup>	[%]	-	-	-	-	-	-
Al <sub>2</sub> O <sub>3</sub> <sup>3 or 6</sup>	[%]	10,0 <sup>3</sup>	10,0 <sup>3</sup>	10,0 <sup>6</sup>	15,0 <sup>3</sup>	-	-
Al <sub>2</sub> O <sub>3</sub> + 1%Pt <sup>7</sup>	[%]	-	-	-	-	10,0	15,0
Al <sub>2</sub> O <sub>3</sub> + 1%(Pd+Pt) <sup>8</sup>	[%]	-	-	-	-	-	-
D = Iron(III)oxide <sup>9</sup>	[%]	-	-	-	-	-	-
Aluminiumsilicate <sup>10</sup>	[%]	-	-	-	-	-	-
Siliconnitride Si <sub>3</sub> N <sub>4</sub>	[%]	-	-	-	-	-	-
E = Graphite	[%]	-	0,5	0,5	-	-	-
Methylhydroxyethylcellulose <sup>11</sup>	[%]	-	-	-	-	-	-
Polyvinylbutyrale	[%]	-	-	-	-	-	-
Calculated Values:							
Gas yield (V=constant)	[mol/kg]	24,3	22,8	22,8	22,4	24,3	22,4
Temperature (p=135*10 <sup>5</sup> Pa)	[K]	2050	2380	2380	2330	2430	2330
Measured Values (in 60 dm <sup>3</sup> can):							
Carbonmonoxide	[ppm]	5700	6000	8000	5000	4600	4200
Nitrogenoxide	[ppm]	300	450	600	300	200	250
Ammonia	[ppm]	0	0	0	0	0	0
Coarse dust in the can	[g]	1,0	0,7	0,8	0,3	1,2	0,5
Fine dust in the can	[g]	0,4	0,1	0,3	0,3	0,3	0,3

Example No.		25	26	27	28	29	30
A = ATZ	[%]	-	-	-	-	-	-
NIGU	[%]	50,6	46,5	43,5	37,4	48,0	-
Ca-DCA	[%]	-	-	-	-	-	-
Na-DCA	[%]	-	-	-	-	-	-
TAGN	[%]	-	-	-	-	-	-
GuNO <sub>3</sub>	[%]	-	-	-	-	-	51,7
B = KNO <sub>3</sub>	[%]	39,4	-	45,9	-	41,4	-
Sr(NO <sub>3</sub> ) <sub>2</sub>	[%]	-	38,5	-	52,1	-	37,8
NaNO <sub>3</sub>	[%]	-	-	-	-	-	-
C = TiO <sub>2</sub> <sup>1</sup>	[%]	-	-	-	-	-	-
ZrO <sub>2</sub> <sup>2</sup>	[%]	-	-	-	-	-	-
Al <sub>2</sub> O <sub>3</sub> <sup>3</sup>	[%]	-	-	-	-	5,0	5,0
Al <sub>2</sub> O <sub>3</sub> + 1%Pt <sup>7</sup>	[%]	-	-	-	-	-	-
Al <sub>2</sub> O <sub>3</sub> + 1%(Pd+Pt) <sup>8</sup>	[%]	10,0	15,0	-	-	-	-
D = Iron(III)oxide <sup>9</sup>	[%]	-	-	5,0	-	5,0	5,0
Aluminiumsilicate <sup>10</sup>	[%]	-	-	-	-	-	-
Siliconnitride Si <sub>3</sub> N <sub>4</sub>	[%]	-	-	5,0	10,0	-	-
E = Graphite	[%]	-	-	0,6	0,5	0,6	-
Methylhydroxyethylcellulose <sup>11</sup>	[%]	-	-	-	-	-	-
Polyvinylbutyrale	[%]	-	-	-	-	-	0,5
Calculated Values:							
Gas yield (V=constant)	[mol/kg]	24,3	22,4	23,3	19,8	23,6	26,0
Temperature (p=135*10 <sup>5</sup> Pa)	[K]	2430	2330	2130	2820	1970	2100
Measured Values (in 60 dm <sup>3</sup> can):							
Carbonmonoxide	[ppm]	4500	4000	6300	6700	8000	5500
Nitrogenoxide	[ppm]	250	250	400	450	150	900
Ammonia	[ppm]	0	0	0	0	250	10
Coarse dust in the can	[g]	1,1	0,4	1,3	1,3	1,5	0,6
Fine dust in the can	[g]	0,2	0,3	0,4	0,5	0,3	0,4

Example No.		31	32	33	34	35	36
A = ATZ	[%]	-	-	-	-	-	-
NIGU	[%]	-	43,0	17,7	9,0	18,1	16,0
Ca-DCA	[%]	27,8	3,0	17,7	23,8	-	-
Na-DCA	[%]	-	-	-	-	18,1	16,0
TAGN	[%]	-	-	-	-	-	-
GuNO <sub>3</sub>	[%]	-	-	-	-	-	-
B = KNO <sub>3</sub>	[%]	-	-	-	57,2	-	58,0
Sr(NO <sub>3</sub> ) <sub>2</sub>	[%]	62,2	45,5	54,6	-	53,8	-
NaNO <sub>3</sub>	[%]	-	-	-	-	-	-
C = TiO <sub>2</sub> <sup>1</sup>	[%]	-	-	-	-	-	-
ZrO <sub>2</sub> <sup>2</sup>	[%]	-	-	-	-	-	-
Al <sub>2</sub> O <sub>3</sub> <sup>3</sup>	[%]	10,0	8,0	10,0	10,0	10,0	10,0
Al <sub>2</sub> O <sub>3</sub> + 1%Pt <sup>7</sup>	[%]	-	-	-	-	-	-
Al <sub>2</sub> O <sub>3</sub> + 1%(Pd+Pt) <sup>8</sup>	[%]	-	-	-	-	-	-
D = Iron(III)oxide <sup>9</sup>	[%]	-	-	-	-	-	-
Aluminiumsilicate <sup>10</sup>	[%]	-	-	-	-	-	-
Siliconnitride Si <sub>3</sub> N <sub>4</sub>	[%]	-	-	-	-	-	-
E = Graphite	[%]	-	-	-	-	-	-
Methylhydroxyethylcellulose <sup>11</sup>	[%]	-	0,5	-	-	-	-
Polyvinylbutyrale	[%]	-	-	-	-	-	-
Calculated Values:							
Gas yield (V=constant)	[mol/kg]	11,4	22,5	15,8	14,0	17,4	14,7
Temperature (p=135*10 <sup>5</sup> Pa)	[K]	2440	2470	2420	1780	2230	1780
Measured Values (in 60 dm <sup>3</sup> can):							
Carbonmonoxide	[ppm]	2800	8000	3600	8000	10000	450
Nitrogenoxide	[ppm]	700	1000	800	500	800	100
Ammonia	[ppm]	0	0	0	50	3	2
Coarse dust in the can	[g]	2,2	0,6	1,2	3,2	1,3	1,5
Fine dust in the can	[g]	0,5	0,3	0,4	0,4	0,2	0,3



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Example No.		43	44	45	46	47	48
A = ATZ	[%]	17,7	-	-	-	-	-
NIGU	[%]	-	-	-	-	-	-
Ca-DCA	[%]	-	-	18,8	-	-	-
Na-DCA	[%]	-	-	-	-	-	-
TAGN	[%]	17,7	-	-	-	-	-
GuNO <sub>3</sub>	[%]	-	54,2	18,8	50,0	50,0	51,5
B = KNO <sub>3</sub>	[%]	44,6	35,8	52,4	-	-	-
Sr(NO <sub>3</sub> ) <sub>2</sub>	[%]	-	-	-	39,4	39,4	38,0
NaNO <sub>3</sub>	[%]	-	-	-	-	-	-
C = TiO <sub>2</sub> <sup>1</sup>	[%]	-	-	-	-	10,0	-
ZrO <sub>2</sub> <sup>2</sup>	[%]	-	-	-	-	-	10,0
Al <sub>2</sub> O <sub>3</sub> <sup>3</sup>	[%]	20,0	5,0	10,0	10,0	-	-
Al <sub>2</sub> O <sub>3</sub> + 1%Pt <sup>7</sup>	[%]	-	-	-	-	-	-
Al <sub>2</sub> O <sub>3</sub> + 1%(Pd+Pt) <sup>8</sup>	[%]	-	-	-	-	-	-
D = Iron(III)oxide <sup>9</sup>	[%]	-	5,0	-	-	-	-
Aluminiumsilicate <sup>10</sup>	[%]	-	-	-	-	-	-
Siliconnitride Si <sub>3</sub> N <sub>4</sub>	[%]	-	-	-	-	-	-
E = Graphite	[%]	-	-	-	0,6	0,6	-
Methylhydroxyethylcellulose <sup>11</sup>	[%]	-	-	-	-	-	0,5
Polyvinylbutyrale	[%]	-	-	-	-	-	-
Calculated Values:							
Gas yield (V=constant)	[mol/kg]	20,0	26,6	16,9	25,1	25,1	25,7
Temperature (p=135*10 <sup>5</sup> Pa)	[K]	1810	1780	1780	2120	2130	2170
Measured Values (in 60 dm <sup>3</sup> can):							
Carbonmonoxide	[ppm]	1000	5000	7000	6000	4000	3500
Nitrogenoxide	[ppm]	150	400	150	800	100	500
Ammonia	[ppm]	50	100	150	5	0	10
Coarse dust in the can	[g]	1,0	2,0	1,8	1,5	1,0	0,5
Fine dust in the can	[g]	0,4	0,5	0,6	0,4	0,5	0,3

Example No.		49	50	51	52	53	54
A = ATZ	[%]	29,75	30,2	30,2	26,5	26,8	33,7
	NIGU	-	-	-	8,0	-	-
	Ca-DCA	-	-	-	-	-	-
	Na-DCA	-	-	-	-	-	-
	TAGN	-	-	-	-	-	-
	GuNO <sub>3</sub>	-	-	-	-	8,0	-
B = KNO <sub>3</sub>		[%]	50,25	49,8	49,8	32,5	32,2
Sr(NO <sub>3</sub> ) <sub>2</sub>		[%]	-	-	-	-	-
NaNO <sub>3</sub>		[%]	-	-	-	15,0	15,0
C = TiO <sub>2</sub> <sup>1</sup>		[%]	-	-	-	-	10,0
ZrO <sub>2</sub> <sup>2</sup>		[%]	3,0	10,0	-	-	-
Al <sub>2</sub> O <sub>3</sub> <sup>3</sup>		[%]	14,0	10,0	10,0	18,0	18,0
Al <sub>2</sub> O <sub>3</sub> + 1%Pt <sup>7</sup>		[%]	-	-	-	-	-
Al <sub>2</sub> O <sub>3</sub> + 1%(Pd+Pt) <sup>8</sup>		[%]	3,0	-	-	-	-
D = Iron(III)oxide <sup>9</sup>		[%]	-	-	-	-	-
Aluminiumsilicate <sup>10</sup>		[%]	-	-	10,0	-	-
Siliconnitride Si <sub>3</sub> N <sub>4</sub>		[%]	-	-	-	-	-
E = Graphite		[%]	-	-	-	-	-
Methylhydroxyethylcellulose <sup>11</sup>		[%]	-	-	-	-	-
Polyvinylbutyrale		[%]	-	-	-	-	-
Calculated Values:							
Gas yield (V=constant)		[mol/kg]	17,6	17,8	19,3	19,4	19,7
Temperature (p=135*10 <sup>5</sup> Pa)		[K]	1780	1780	1920	1800	1780
Measured Values (in 60 dm <sup>3</sup> can):							
Carbonmonoxide		[ppm]	2600	3000	4500	3500	6500
Nitrogenoxide		[ppm]	300	200	300	800	500
Ammonia		[ppm]	23	50	50	0	5
Coarse dust in the can		[g]	1,0	1,1	1,2	0,8	1,0
Fine dust in the can		[g]	0,2	0,4	0,5	0,2	0,2

of the *Journal of the American Medical Association* (JAMA) and the *New England Journal of Medicine* (NEJM).

The burn-up tests have been carried out in a practise-orientated housing of a gas generator for a 60 litre driver-airbag having the original size, igniter and filter package of stainless steel.

The weight of the propelling charge for gas generators was 50 to 55 g, depending on the gas yield of the individual composition of the propellant for gas generators.

Depending on the burn-up properties the pellets had a diameter of 4 to 6 mm and a height of 1.5 and 2.1 mm, respectively.

The gas yield and the temperature lie in a range which is advantageous for propellants for gas generators for airbags.

The terms "coarse dust" and "fine dust" in the table refer to the dust in the can after burn-up.

The values measured for CO, NO<sub>x</sub> and NH<sub>3</sub> given in the table relate to a 60 litre can. The obtained values are good taking into consideration that a non-optimised test gas generator has been used.

From the comparison of Examples 14 and 15, 17 and 18, and 20 and 21, the effect obtained by the use of highly dispersed oxides in comparison to the conventional oxides is illustrated. The decrease in particle ejection (coarse and fine dust) for the system nitroguanidine/strontium nitrate has been about 20 to 40% due to the specific highly dispersed slag trap (C) used according to the present invention in comparison to the conventional oxides of the same chemical structural formula but having a lower specific surface. In addition, the reduction of toxic gases of about 10 to 25% due to the improvement of the burn-up by use of the specific slag

traps (C) used according to the present invention and the properties thereof is evident.

Furthermore, the additional beneficial effect of highly dispersed slag traps (C), having catalysts on the surface, with respect to the formation of toxic gases can be seen from the comparison of the propellants for gas generators according to e.g. Examples 2 to 8 and 10.

The portions of CO and NO<sub>x</sub> are according to Examples 8 and 10 (with catalyst) below those of Example 2 (without catalyst, however, apart from that having the same composition).

Particularly preferred compositions are those of Examples 14, 17 and 20.

The thermodynamical data of the various compositions have been calculated with respect to the oxygen-balance-excess for which the smallest possible amount of toxic gases during burn-up has been expected.

CLAIMS:

1. Propellant for gas generators, comprising

(A) at least one fuel selected from the group consisting of guanidine nitrate (GUNI;  $\text{GuNO}_3$ ), dicyanamide, ammonium dicyanamide, sodium dicyanamide (Na-DCA), copper dicyanamide, tin dicyanamide, calcium dicyanamide (Ca-DCA), guanidine dicyanamide (GDCA), aminoguanidine bicarbonate (AGB), aminoguanidine nitrate (AGN), triaminoguanidine nitrate (TAGN), nitroguanidine (NIGU), dicyandiamide (DCD), azodicarbonamide (ADCA) as well as tetrazole (HTZ), 5-aminotetrazole (ATZ), 5-nitro-1,2,4-triazole-3-on (NTO), salts and mixtures thereof,

(B) at least one alkali metal nitrate or alkaline earth metal nitrate or ammonium nitrate, -chlorate or -perchlorate,

(C) at least one essentially chemically-inert slag trap with a high fusion point, selected from the group comprising  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$  and  $\text{ZrO}_2$  in highly dispersed form or mixtures thereof.

2. Propellant for gas generators according to claim 1, wherein component (A) is present in an amount of about 20 to 60 wt.-%, preferably of about 28 to 52 wt.-% and in particular of about 45 to 51 wt.-%, component (B) is present in an amount of about 38 to about 63 wt.-%, preferably of about 38 to about 55 wt.-% and in particular of about 39 to 45 wt.-%, component (C) is present in an amount of about 5 to 22 wt.-%, preferably of about 8 to 20 wt.-% and in particular of about 9 to 11 wt.-%.

3. Propellant for gas generators according to claim 1 or 2, wherein component (A) is selected from the group consist-

ing of nitroguanidine, 5-aminotetrazole, dicyandiamide, dicyanamide, sodium- and calcium dicyanamide and guanidine nitrate, and mixtures thereof.

4. Propellant for gas generators according to any one of claims 1 to 3, wherein component (B) is selected from the group consisting of sodium-, potassium- and strontium nitrate.

5. Propellant for gas generators according to any one of claims 1 to 4, wherein component (C) is selected from the group consisting of highly dispersed  $\text{Al}_2\text{O}_3$ , highly dispersed  $\text{TiO}_2$  and highly dispersed  $\text{ZrO}_2$ .

6. Propellant for gas generators according to claim 5, wherein component (C) is selected from the group consisting of highly dispersed  $\text{Al}_2\text{O}_3$  having a specific surface of  $100 \pm 15 \text{ m}^2/\text{g}$ , highly dispersed  $\text{TiO}_2$  having a specific surface of  $50 \pm 15 \text{ m}^2/\text{g}$  and highly dispersed  $\text{ZrO}_2$  having a specific surface of  $40 \pm 10 \text{ m}^2/\text{g}$ .

7. Propellant for gas generators according to claim 5, wherein a part of the component (C) is a carrier for a platinum metal or a metal alloy of platinum metals or copper in a catalytic effective layer thickness.

8. Propellant for gas generators according to claim 7, wherein the platinum metal is selected from ruthenium (Ru), Osmium (Os), rhodium (Rh), iridium (Ir), palladium (Pd) and platinum (Pt).

9. Propellant for gas generators according to claim 7, wherein the metal alloy of platinum metals is selected from Pt/Pd and Pt/Rh alloys.

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10. Propellant for gas generators according to any one of claims 7 to 9, wherein the weight portion of the catalyst with respect to component (C) is 0.1 to 5 wt.-%, preferably 0.2 to 1.2 wt.-%.

11. Propellant for gas generators according to any one of claims 1 to 10, wherein component (A) is nitroguanidine, component (B) is strontium nitrate and component (C) is highly dispersed  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$  or  $\text{ZrO}_2$ .

12. Propellant for gas generators according to claim 11, wherein component (A) is present in an amount of 45 to 51 wt.-%, component (B) is present in an amount of 39 to 45 wt.-% and component (C) is present in an amount of 9 to 11 wt.-%, with respect to the total composition.

13. Propellant for gas generators according to any one of claims 1 to 11, containing in addition component (D) at least one slag former, selected from alkali metal and alkaline earth metal carbonates, alkali metal and alkaline earth metal oxides, silicates, aluminates, aluminium silicates, silicon nitride ( $\text{Si}_3\text{N}_4$ ) and iron(III)oxide.

14. Propellant for gas generators according to claim 13, wherein component (D) is present in an amount of about 2 to 12 wt.-%, preferably in an amount of about 4 to 10 wt.-%.

15. Propellant for gas generators according to any one of claims 1 to 14, further containing component (E) at least one binder being soluble in water at room temperature.

16. Propellant for gas generators according to claim 15, wherein the binder is selected from the group consisting of cellulose compounds, polymers of one or more polymerisable

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olefinic unsaturated monomers, a metal salt of stearic acid being insoluble in water at room temperature and graphite.

17. Propellant for gas generators according to claim 15 or 16, wherein the binder is present in an amount of 0 to 2 wt.-%, preferably of 0.3 to 0.8 wt.-%.

18. Use of the propellant for gas generators according to any one of claims 1 to 17 as gas-generating agent in airbags, as extinguishing agent or as propellant.

AMENDED CLAIMS:

## 1. Propellant for gas generators, comprising

(A) at least one fuel selected from the group consisting of guanidine nitrate (GUNI;  $\text{GuNO}_3$ ), dicyanamide, ammonium dicyanamide, sodium dicyanamide (Na-DCA), copper dicyanamide, tin dicyanamide, calcium dicyanamide (Ca-DCA), guanidine dicyanamide (GDCA), aminoguanidine bicarbonate (AGB), aminoguanidine nitrate (AGN), triaminoguanidine nitrate (TAGN), nitroguanidine (NIGU), dicyandiamide (DCD), azodicarbonamide (ADCA) as well as tetrazole (HTZ), 5-aminotetrazole (ATZ), 5-nitro-1,2,4-triazole-3-on (NTO), salts and mixtures thereof,

(B) at least one alkali metal nitrate or alkaline earth metal nitrate or ammonium nitrate, -chlorate or -perchlorate,

(C) at least one essentially chemically-inert slag trap with a high fusion point, selected from the group comprising highly dispersed  $\text{Al}_2\text{O}_3$  having a specific surface of  $100 \pm 15 \text{ m}^2/\text{g}$ , highly dispersed  $\text{TiO}_2$  having a specific surface of  $50 \pm 15 \text{ m}^2/\text{g}$  and highly dispersed  $\text{ZrO}_2$  having a specific surface of  $40 \pm 10 \text{ m}^2/\text{g}$  and mixtures thereof.

2. Propellant for gas generators according to claim 1, wherein component (A) is present in an amount of about 20 to 60 wt.-%, preferably of about 28 to 52 wt.-% and in particular of about 45 to 51 wt.-%, component (B) is present in an amount of about 38 to about 63 wt.-%, preferably of about 38 to about 55 wt.-% and in particular of about 39 to 45 wt.-%, component (C) is present in an amount of about 5 to 22 wt.-%,

preferably of about 8 to 20 wt.-% and in particular of about 9 to 11 wt.-%.

3. Propellant for gas generators according to claim 1 or 2, wherein component (A) is selected from the group consisting of nitroguanidine, 5-aminotetrazole, dicyandiamide, dicyanamide, sodium- and calcium dicyanamide and guanidine nitrate, and mixtures thereof.

4. Propellant for gas generators according to any one of claims 1 to 3, wherein component (B) is selected from the group consisting of sodium-, potassium- and strontium nitrate.

5. Propellant for gas generators according to any one of claims 1 to 4, wherein a part of the component (C) is a carrier for a platinum metal or a metal alloy of platinum metals or copper in a catalytic effective layer thickness.

6. Propellant for gas generators according to claim 5, wherein the platinum metal is selected from ruthenium (Ru), Osmium (Os), rhodium (Rh), iridium (Ir), palladium (Pd) and platinum (Pt).

7. Propellant for gas generators according to claim 5, wherein the metal alloy of platinum metals is selected from Pt/Pd and Pt/Rh alloys.

8. Propellant for gas generators according to any one of claims 5 to 7, wherein the weight portion of the catalyst with respect to component (C) is 0.1 to 5 wt.-%, preferably 0.2 to 1.2 wt.-%.

9. Propellant for gas generators according to any one of claims 1 to 8, wherein component (A) is nitroguanidine, component (B) is strontium nitrate and component (C) is highly dispersed  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$  or  $\text{ZrO}_2$ .

10. Propellant for gas generators according to claim 9, wherein component (A) is present in an amount of 45 to 51 wt.-%, component (B) is present in an amount of 39 to 45 wt.-% and component (C) is present in an amount of 9 to 11 wt.-%, with respect to the total composition.

11. Propellant for gas generators according to any one of claims 1 to 9, containing in addition component (D) at least one slag former, selected from alkali metal and alkaline earth metal carbonates, alkali metal and alkaline earth metal oxides, silicates, aluminates, aluminium silicates, silicon nitride ( $\text{Si}_3\text{N}_4$ ) and iron(III)oxide.

12. Propellant for gas generators according to claim 11, wherein component (D) is present in an amount of about 2 to 12 wt.-%, preferably in an amount of about 4 to 10 wt.-%.

13. Propellant for gas generators according to any one of claims 1 to 12, further containing component (E) at least one binder being soluble in water at room temperature.

14. Propellant for gas generators according to claim 13, wherein the binder is selected from the group consisting of cellulose compounds, polymers of one or more polymerisable olefinic unsaturated monomers, a metal salt of stearic acid being insoluble in water at room temperature and graphite.



15. Propellant for gas generators according to claim 13 or 14, wherein the binder is present in an amount of 0 to 2 wt.-%, preferably of 0.3 to 0.8 wt.-%.

16. Use of the propellant for gas generators according to any one of claims 1 to 15 as gas-generating agent in airbags, as extinguishing agent or as propellant.

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Docket No.  
15268.1

## Declaration and Power of Attorney For Patent Application

### English Language Declaration

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled  
**PROPELLANTS FOR GAS GENERATOR**

the specification of which

(check one)

☐ is attached hereto,

☒ was filed on September 19, 2000 as United States Application No. or PCT International Application Number 09/466,767 and was amended on \_\_\_\_\_ (if applicable)

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, Section 119(a)-(d) or Section 365(b) of any foreign application(s) for patent or inventor's certificate, or Section 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate or PCT International application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application(s)

Priority Not Claimed

198 12 372.8

(Number)

Germany

(Country)

20 March 1988

(Day/Month/Year Filed)

☐

(Number)

(Country)

(Day/Month/Year Filed)

☐

(Number)

(Country)

(Day/Month/Year Filed)

☐

I hereby claim the benefit under 35 U.S.C. Section 119(e) of any United States provisional application(s) listed below:

(Application Serial No.)

(Filing Date)

(Application Serial No.)

(Filing Date)

(Application Serial No.)

(Filing Date)

I hereby claim the benefit under 35 U. S. C. Section 120 of any United States application(s), or Section 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. Section 112, I acknowledge the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, C. F. R., Section 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application;

(Application Serial No.)

(Filing Date)

(Status)  
(patented, pending, abandoned)

(Application Serial No.)

(Filing Date)

(Status)  
(patented, pending, abandoned)

(Application Serial No.)

(Filing Date)

(Status)  
(patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.



POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith. (list name and registration number)

Send Correspondence to:

**John M. Guyonn**  
**Attorney for Applicants**  
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**022913**

PATENT TRADEMARK OFFICE

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Full name of sixth inventor, if any

Sixth inventor's signature

Date

Residence

Citizenship

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